

~~102044~~  
=> d his

(FILE 'HOME' ENTERED AT 09:41:10 ON 17 AUG 2005)

FILE 'REGISTRY' ENTERED AT 09:41:47 ON 17 AUG 2005

L1           STRUCTURE UPLOADED  
L2           0 S L1 CSS  
L3           43 S L1  
L4           1056 S L1 FUL

FILE 'CAPLUS' ENTERED AT 09:43:18 ON 17 AUG 2005

L5           516 S L4  
L6           102044 S DIABETES  
L7           11 S L6 AND L5  
L8           STRUCTURE UPLOADED  
              S L8

FILE 'REGISTRY' ENTERED AT 09:47:30 ON 17 AUG 2005

L9           25 S L8

FILE 'CAPLUS' ENTERED AT 09:47:30 ON 17 AUG 2005

L10          29 S L9

FILE 'REGISTRY' ENTERED AT 09:47:38 ON 17 AUG 2005

L11          663 SEARCH L8 SSS   SUB=L4 FUL

FILE 'CAPLUS' ENTERED AT 09:48:13 ON 17 AUG 2005

L12          338 S L11  
L13          11 S L12 AND L6

FILE 'REGISTRY' ENTERED AT 09:48:58 ON 17 AUG 2005

L14          15 S L8 CSS FUL

FILE 'CAPLUS' ENTERED AT 09:49:52 ON 17 AUG 2005

L15          58 S L14

FILE 'USPATFULL' ENTERED AT 09:50:01 ON 17 AUG 2005

L16          4 S L15

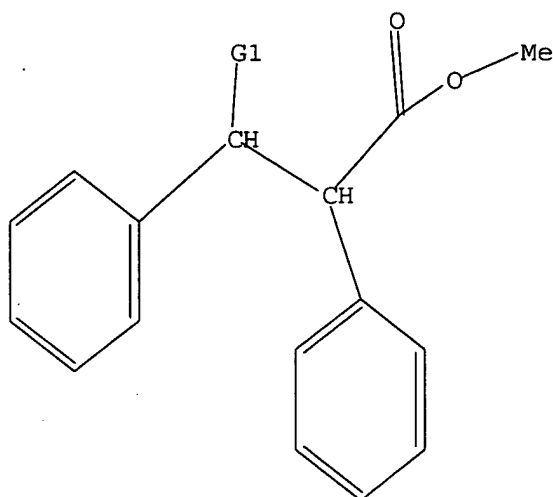
FILE 'CAPLUS' ENTERED AT 09:51:01 ON 17 AUG 2005

=> d l8

L8 HAS NO ANSWERS

L8           STR

10/446,319



G1 H, Me, Et, n-Pr, i-Pr, n-Bu, i-Bu, OH

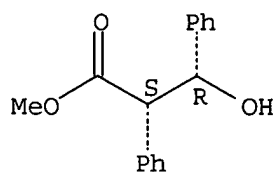
Structure attributes must be viewed using STN Express query preparation.

=> d bib abs hitstr 35-58 115

L15 ANSWER 35 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1982:169662 CAPLUS  
DN 96:169662  
TI Effect of shift reagents on infrared spectra  
AU Porter, Geoffrey B.; Simpson, Jon  
CS Dep. Chem. Metall., R. Mil. Coll. Sci., Shrivenham/Swindon/Wilts., SN6  
81A, UK  
SO Spectroscopy Letters (1981), 14(11-12), 755-61  
CODEN: SPLEBX; ISSN: 0038-7010  
DT Journal  
LA English  
AB The IR spectra of Eu(fod)<sub>3</sub> shift reagent (Hfod = 6,6,7,7,8,8,8-heptafluoro-2,2-dimethyl-3,5-octanedione) were determined in CCl<sub>4</sub> solns. containing Me threo- and erythro-2,3-diphenyl-3-hydroxypropanoate, Me 2-phenylethanoate, and 2-butanone at 25°. An adsorption at 1690 cm<sup>-1</sup> is due to CO-Eu interaction. Equilibrium consts. were calculated from peak areas by assuming 1:1 interaction. The pK values are 3.7, 3.6, 2.4, and 2.9 ± 0.1, resp. The OH group reinforces CO interaction via a chelate effect.  
IT **17226-93-6DP**, europium shift reagent complexes  
**17226-94-7DP**, europium shift reagent complexes  
RL: FORM (Formation, nonpreparative); PREP (Preparation)  
(formation of, in carbon tetrachloride, IR spectral study of)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid, β-hydroxy-α-phenyl-, methyl ester,  
(αR,βS)-rel- (9CI) (CA INDEX NAME)

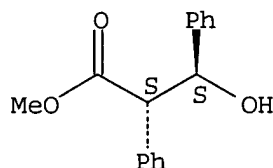
Relative stereochemistry.

10/446,319



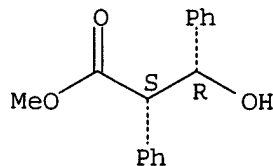
RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 36 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1980:532206 CAPLUS  
DN 93:132206  
TI Effect of the type of base on the yield and stereoselectivity of the  
low-temperature Claisen reaction. II. Experiments with alkali metals  
AU Krachanov, Kh.; Kirchev, N.; Kurtev, B.  
CS Vissh Inst. Khranitel. Vkusova Prom., Plovdiv, Bulg.  
SO Nauchni Trudove - Vissh Institut po Khranitelna i Vkusova Promishlenost,  
Plovdiv (1978), 25, Pt. 1, 333-5  
CODEN: NTKVAH; ISSN: 0477-0250  
DT Journal  
LA Bulgarian  
AB The catalytic activity of alkali metals in Claisen condensation of  
PhCH<sub>2</sub>CO<sub>2</sub>Me with PhCHO increased in the order Li  $\ll$  Na < K.  
HOCHPhCHPhCO<sub>2</sub>Me was formed in 72 and 84% yield and 4:1 and 2.8:1  
threo-erythro ratio with Na and K, resp.  
IT **17226-93-6P 17226-94-7P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

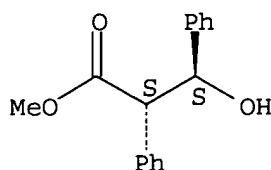
Relative stereochemistry.



RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

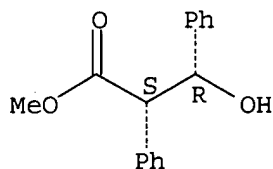
Relative stereochemistry.

10/446,319



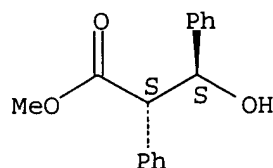
L15 ANSWER 37 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1980:76060 CAPLUS  
DN 92:76060  
TI Stereoselective synthesis of  $\beta$ -hydroxy esters by low-temperature Claisen reaction. IV. Effect of base on the yield and stereoselectivity of the reaction in ether  
AU Krachanov, Kh.; Kirchev, N.; Kurtev, B.  
CS Vissh Inst. Khranitel. Vkusova Prom., Plovdiv, Bulg.  
SO Izvestiya po Khimiya (1979), 12(1), 3-7  
CODEN: IZKHDX; ISSN: 0324-0401  
DT Journal  
LA German  
AB The Claisen condensation of BzH with  $\text{PhCH}_2\text{CO}_2\text{Me}$  (I) was studied in ether at  $-24^\circ$  in the presence alkali metal amides and ethoxides. The best yields and stereoselectivity for threo-HOCHPhCHPhCO<sub>2</sub>Me were achieved using  $\text{NaNH}_2$  in amts. equivalent to that of I.  
IT **17226-93-6P 17226-94-7P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

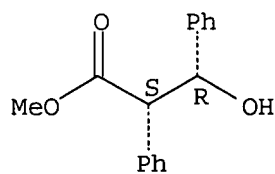
Relative stereochemistry.



L15 ANSWER 38 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1978:529206 CAPLUS  
DN 89:129206

TI Interaction of substituted benzaldehydes with methylphenylacetate during low-temperature Claisen reaction  
 AU Kirchev, N.; Krachanov, Kh.  
 CS Inst. Food Technol., Plovdiv, Bulg.  
 SO Doklady Bolgarskoi Akademii Nauk (1978), 31(1), 59-61  
 CODEN: DBANAD; ISSN: 0366-8681  
 DT Journal  
 LA English  
 AB Reaction of  $\text{PhCH}_2\text{CO}_2\text{Me}$  with  $\text{RnC}_6\text{H}_5\text{-nCHO}$  ( $\text{Rn} = \text{H}, 2\text{-F}, 2\text{-Cl}, 3\text{-Cl}, 2,6\text{-Cl}_2, 3\text{-Me}, 4\text{-MeO}$ , etc.) in  $\text{Et}_2\text{O}$  at  $-24^\circ$  for 2 h in the presence of  $\text{NaNH}_2$  stopped at the aldol stage and gave eighteen  $\text{RnC}_6\text{H}_5\text{-nCH(OH)CHPhCO}_2\text{Me}$  (I) in 31-85% yield, with threo/erythro ratio in the product varying from 96:4 to 69:31. There was no well-defined relation between the nature and position of the substituent and the yield of I.  
 IT **17226-93-6P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 17226-93-6 CAPLUS  
 CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha\text{R},\beta\text{S}$ )-rel- (9CI) (CA INDEX NAME)

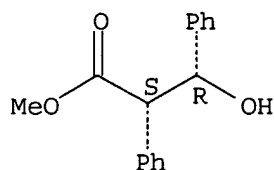
Relative stereochemistry.



L15 ANSWER 39 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1978:104853 CAPLUS  
 DN 88:104853  
 TI Stability of diastereomeric 2,3-diphenyl-3-hydroxypropanoic acids and their esters during heating in triethylamine medium  
 AU Kirchev, N.; Krachanov, Kh.  
 CS Vissh Inst. Khranit. Vkusova Prom., Plovdiv, Bulg.  
 SO Nauchni Trudove - Vissh Institut po Khranitelna i Vkusova Promishlenost, Plovdiv (1976), 23(3), 139-46  
 CODEN: NTKVAH; ISSN: 0477-0250  
 DT Journal  
 LA Bulgarian  
 AB Diastereoisomeric  $\text{ROCHPhCHPhCO}_2\text{R}_1$  (I;  $\text{R} = \text{R}_1 = \text{H}$ ;  $\text{R} = \text{H}, \text{Ac}$ ,  $\text{R}_1 = \text{Me}$ ) were stable toward  $\text{Et}_3\text{N}$  at room temperature in the presence or absence of  $(\text{Me}_2\text{N})_3\text{PO}$ , but underwent retro-aldol decomposition at  $80^\circ$  in the presence of  $(\text{Me}_2\text{N})_3\text{PO}$ . The stability of the acid exceeded that of the esters. No significant isomerization of I was observed. Condensation reaction of  $\text{PhCH}_2\text{CO}_2\text{Me}$  with  $\text{PhCHO}$  in the presence of  $\text{Et}_3\text{N}$  at  $-24$  to  $+80^\circ$  afforded threo- and erythro-I ( $\text{R} = \text{H}$ ,  $\text{R}_1 = \text{Me}$ ) in 1:2 ratio and  $\leq 5\%$  combined yield.  
 IT **17226-93-6 17226-94-7**  
 RL: PRP (Properties)  
 (stability of, to isomerization and retro-aldol decomposition)  
 RN 17226-93-6 CAPLUS  
 CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha\text{R},\beta\text{S}$ )-rel- (9CI) (CA INDEX NAME)

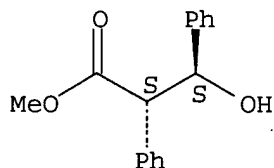
Relative stereochemistry.

10/446,319



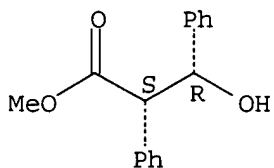
RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 40 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1978:67547 CAPLUS  
DN 88:67547  
TI Effect of shift reagents on NMR coupling constants  
AU Porter, Geoffrey Brian; Simpson, Jon  
CS Chem. Branch, R. Mil. Coll. Sci., Shrivenham/Swindon, UK  
SO Angewandte Chemie (1978), 90(1), 51-2  
CODEN: ANCEAD; ISSN: 0044-8249  
DT Journal  
LA German  
AB A significant effect is reported of the NMR shift reagents [D27]-Eu(fod)<sub>3</sub> and [D27]-La(fod)<sub>3</sub> (Hfod = F<sub>3</sub>C(CF<sub>2</sub>)<sub>2</sub>COCH<sub>2</sub>COCMe<sub>3</sub>) on the J values for vicinal coupling of esters of 2,3-diphenyl-3-hydroxypropionic acid in <sup>1</sup>H NMR. After the addition of the shift reagents the chemical shift of the <sup>1</sup>H NMR signal indicates the formation of a 1:1 complex. The data can be interpreted in terms of chelate formation of the potentially 2-coordinating hydroxy esters, whereby both functional groups are in the gauche position.  
IT **17226-93-6 17226-94-7**  
RL: PRP (Properties)  
(NMR coupling consts. of, lanthanide shift reagent effects on)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

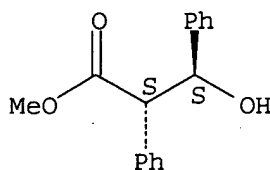
Relative stereochemistry.



RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

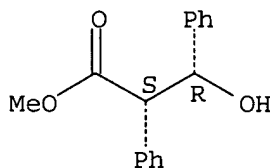
10/446,319

Relative stereochemistry.



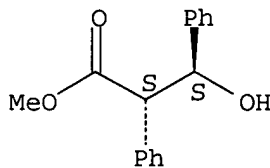
L15 ANSWER 41 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1977:105406 CAPLUS  
DN 86:105406  
TI Stereoselective synthesis of  $\beta$ -hydroxy esters via the low-temperature Claisen reaction  
AU Krachanov, Kh.; Kirchev, N.; Kurtev, B.  
CS Higher Inst. Food Technol., Plovdiv, Bulg.  
SO Izvestiya po Khimiya (1976), 9(2), 338-47  
CODEN: IZKHDX; ISSN: 0324-0401  
DT Journal  
LA English  
AB The condensation of PhCHO with PhCH<sub>2</sub>CO<sub>2</sub>R (R = Me, Et, Pr, CHMe<sub>2</sub>, Bu, CMe<sub>3</sub>) in Et<sub>2</sub>O at low temps. (.apprx. -20°) in the presence of NaNH<sub>2</sub> gave 784% of the threo isomer of PhCH(OH)CHPhCO<sub>2</sub>R. The stereoselectivity is under thermodyn. control. Catalytic amts. of NaNH<sub>2</sub> lower the yields and stereoselectivity slightly.  
IT **17226-93-6P 17226-94-7P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester, ( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.

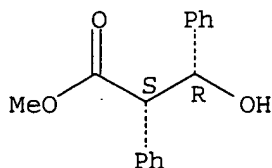


L15 ANSWER 42 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

10/446,319

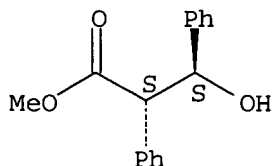
AN 1976:477455 CAPLUS  
DN 85:77455  
TI Effect of substituents on the stereochemistry of the Reformatskii reaction  
AU Mladenova, M.; Blagoev, B.; Kurtev, B.  
CS Inst. Org. Chem., Sofia, Bulg.  
SO Doklady Bolgarskoi Akademii Nauk (1975), 28(12), 1633-6  
CODEN: DBANAD; ISSN: 0366-8681  
DT Journal  
LA French  
AB The Reformatskii reaction of  $\text{RC}_6\text{H}_4\text{CHO}$  ( $\text{R} = \text{H}, \text{p-Me}, \text{o-Me}, \text{p-Cl}, \text{o-Cl}, \text{p-MeO}$ ) and 1-naphthaldehyde with  $\text{p-R}_1\text{C}_6\text{H}_4\text{CHBrCO}_2\text{Me}$  ( $\text{R}_1 = \text{Br}, \text{H}$ ) gave an .apprx.50:50 mixture of erythro- and threo- $\text{RC}_6\text{H}_4\text{CH}(\text{OH})\text{CH}(\text{C}_6\text{H}_4\text{R}_1\text{-p})\text{CO}_2\text{Me}$  or the 1-naphthyl analog in  $\text{Et}_2\text{O}$ . In  $(\text{MeO})_2\text{CH}_2$ , the erythro isomer was slightly favored (.apprx.60:40); in  $\text{Me}_2\text{SO}$ , the threo isomer was favored (.apprx.70:30). In  $\text{Me}_2\text{SO}$ ,  $\text{p-R}_1\text{C}_6\text{H}_4\text{CH}(\text{CO}_2\text{Me})\text{CH}(\text{CO}_2\text{Me})\text{C}_6\text{H}_4\text{R-p}$  was also formed. The lack of substituent effects in the Reformatskii reaction was explained by a transition state resembling the starting materials.  
IT **17226-93-6P 17226-94-7P**  
RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)  
RN 17226-93-6 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha\text{R}, \beta\text{S}$ )-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



RN 17226-94-7 CAPLUS  
CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha\text{R}, \beta\text{R}$ )-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 43 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1976:121375 CAPLUS  
DN 84:121375  
TI Absolute configurations of the diastereomeric 3-hydroxy-2,3-diphenylpropanoic acids  
AU Berova, N.; Kurtev, B.  
CS Inst. Org. Chem., Sofia, Bulg.  
SO Izvestiya po Khimiya (1975), 8(1), 77-83  
CODEN: IZKHDX; ISSN: 0324-0401  
DT Journal  
LA English  
AB Chemical correlation with enantiomeric Me 2,3-diphenylpropanoates showed that



10/446,319

(-)-erythro- and (+)-threo-3-hydroxy-2,3-diphenylpropionic acid have the S,S and 2R,3S configurations, resp.

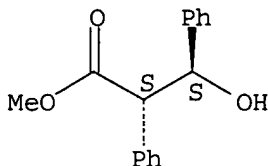
IT 58769-52-1P 58769-53-2P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(preparation and chlorination of)

RN 58769-52-1 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
[S-(R\*,R\*)]- (9CI) (CA INDEX NAME)

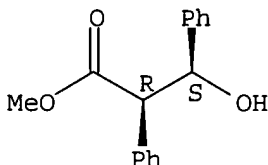
Absolute stereochemistry.



RN 58769-53-2 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
[S-(R\*,S\*)]- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



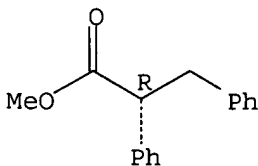
IT 58714-10-6P 58714-11-7P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 58714-10-6 CAPLUS

CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester, ( $\alpha$ R)- (9CI)  
(CA INDEX NAME)

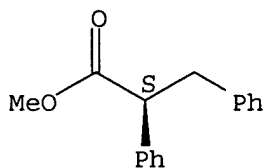
Absolute stereochemistry. Rotation (-).



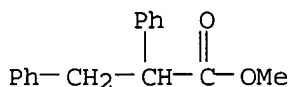
RN 58714-11-7 CAPLUS

CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester, ( $\alpha$ S)- (9CI)  
(CA INDEX NAME)

Absolute stereochemistry.



- L15 ANSWER 44 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1976:89550 CAPLUS  
 DN 84:89550  
 TI Diacids and  $\alpha,\alpha$ -disubstituted mixed functional acids and their derivatives. XLIV-Chain-ring isomerism of some  $\gamma$ -aldehydic acids and disubstituted  $\alpha$ -gem-succinic aldehyde esters. Application to the selective reaction of nucleophiles  
 AU Des Abbayes, Herve; Neveu, Cecile; Salmon-Legagneur, Francois  
 CS Dep. Phys. Crist. Chim. Struct., Univ. Rennes, Rennes, Fr.  
 SO Bulletin de la Societe Chimique de France (1973), (9-10, Pt. 2), 2686-92  
 CODEN: BSCFAS; ISSN: 0037-8968  
 DT Journal  
 LA French  
 OS CASREACT 84:89550  
 GI For diagram(s), see printed CA Issue.  
 AB The open and closed isomers,  $\text{HCOCH}_2\text{CRR}_1\text{CO}_2\text{H}$  (I,  $\text{R} = \text{R}_1 = \text{Me}$ ;  $\text{R} = \text{Ph}$ ,  $\text{R}_1 = \text{Me}$ ,  $\text{Ph}$ ,  $\text{PhCH}_2$ ) and II ( $\text{R}$ ,  $\text{R}_1$  have same meaning), from the ring-chain isomerization of I were blocked by methylation, which gave the Me ester (III) of I and the O-Me derivative (IV) of II and permitted selective reaction with nucleophiles. Thus, I were quant. converted by  $\text{MeMgI}$  into valerolactones (V,  $\text{R}_2 = \text{Me}$ ) (VI); VI were also obtained, in some cases, by the reaction of  $\text{MeMgI}$  with III, but reaction of  $\text{MeMgI}$  with IV gave  $\text{MeOCHMeCH}_2\text{CRR}_1\text{CO}_2\text{H}$ . Reaction of I with morpholine gave V ( $\text{R}_2 = \text{morpholino}$ ), III gave ester enamines  $\text{R}_2\text{NCH:CHCRR}_1\text{CO}_2\text{Me}$  ( $\text{R}$ ,  $\text{R}_1$  same as in I,  $\text{R}_2\text{N} = \text{morpholino}$ ), and IV did not react. The mechanisms of the reactions are discussed.  
 IT **35030-49-0**  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (reaction of, with bromoacetaldehyde acetal)  
 RN 35030-49-0 CAPLUS  
 CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester (9CI) (CA INDEX NAME)



- L15 ANSWER 45 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1975:125008 CAPLUS  
 DN 82:125008  
 TI Application of the low temperature Claisen reaction for stereoselective synthesis of threo-3-aryl-3-hydroxy-2-phenylpropanoic acids and their methyl esters  
 AU Kurtev, B.; Kratchanov, Kh.; Kirchev, N.  
 CS Inst. Org. Chem., Sofia, Bulg.  
 SO Synthesis (1975), (2), 106-8  
 CODEN: SYNTBF; ISSN: 0039-7881  
 DT Journal  
 LA English

10/446,319

OS CASREACT 82:125008

AB RCHO (R = Ph, 4-FC<sub>6</sub>H<sub>4</sub>, 2-, 3-, 4-ClC<sub>6</sub>H<sub>4</sub>, 2,6-Cl<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 2- and 4-BrC<sub>6</sub>H<sub>4</sub>) condensed with PhCH<sub>2</sub>CO<sub>2</sub>R<sub>1</sub> (R<sub>1</sub> = Me, CMe<sub>3</sub>) at -24° in Et<sub>2</sub>O or (Me<sub>2</sub>CH)<sub>2</sub>O containing NaNH<sub>2</sub> gave threo-HOCH<sub>2</sub>CHPhCO<sub>2</sub>R<sub>1</sub> (I) in 40-85% yield from the solid phase of the reaction mixture; I (R = Ph, R<sub>1</sub> = CM<sub>3</sub>) was hydrolyzed to I (R = Ph, R<sub>1</sub> = H) in 93% yield by heating with CF<sub>3</sub>CO<sub>2</sub>H. The I yield was lower and the erythro-threo ratio was higher in different solvents or with NaOEt instead of NaNH<sub>2</sub>.

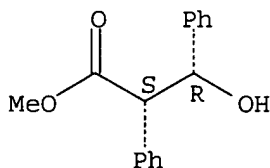
IT 17226-93-6P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 46 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1975:30853 CAPLUS

DN 82:30853

TI Stereochemistry of the Reformatsky reaction

AU Mladenova, M.; Blagoev, B.; Kurtev, B.

CS Inst. Org. Chim., Sofia, Bulg.

SO Bulletin de la Societe Chimique de France (1974), 7-8, Pt. 2, 1464-8

CODEN: BSCFAS; ISSN: 0037-8968

DT Journal

LA French

AB The effect of solvent, temperature, alkyl group (R) and metal (Zn, Mg) on the stereochem. and the reversibility of the Reformatskii reaction of PhCHBrCO<sub>2</sub>R (R = Me, CHMe<sub>2</sub> and CMe<sub>3</sub>) with BzH was investigated.

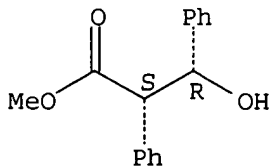
IT 17226-93-6P 17226-94-7P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

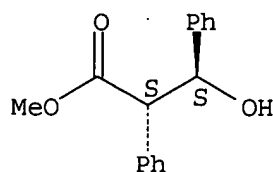
Relative stereochemistry.



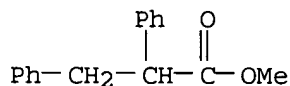
RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

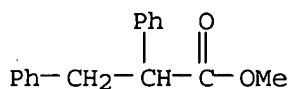
Relative stereochemistry.



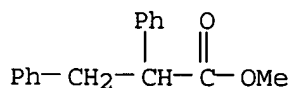
L15 ANSWER 47 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1974:463032 CAPLUS  
 DN 81:63032  
 TI NMR spectra and conformation of 1,2-diphenylpropane and 2,3-diphenylpropanoic acid  
 AU Spassov, S. L.; Orahovats, A. S.; Mishev, S. M.; Schraml, J.  
 CS Inst. Org. Chem., Bulg. Acad. Sci., Sofia, Bulg.  
 SO Tetrahedron (1974), 30(2), 365-8  
 CODEN: TETRAB; ISSN: 0040-4020  
 DT Journal  
 LA English  
 AB NMR spectra of PhCH<sub>2</sub>CHPhR ( = R Me, CO<sub>2</sub>H, CO<sub>2</sub>Me) and deuterated analogs showed the predominance of the conformer with anti-periplanar Ph groups. The values of the gauche nonbonded interaction energies (-ΔE kcal/mole) are 0.8 for Ph/Ph, 0.6 for Ph/CO<sub>2</sub>H, and 0.4 for Ph/ CO<sub>2</sub>Me.  
 IT **35030-49-0**  
 RL: PRP (Properties)  
 (conformation of, NMR in relation to)  
 RN 35030-49-0 CAPLUS  
 CN Benzenepropanoic acid, α-phenyl-, methyl ester (9CI) (CA INDEX NAME)



L15 ANSWER 48 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1973:485283 CAPLUS  
 DN 79:85283  
 TI Constitution dependence and the benzene induced effect of the chemical shifts of some carboxylic acid methyl ester methoxy protons  
 AU Brink, Maud; Larsson, Erik  
 CS Chem. Inst., Univ. Lund, Lund, Swed.  
 SO Organic Magnetic Resonance (1973), 5(7), 327-31  
 CODEN: ORMRED; ISSN: 0030-4921  
 DT Journal  
 LA English  
 AB The chemical shifts of the MeO protons in .apprx.60 Me esters of carboxylic acids were obtained in CCl<sub>4</sub> and C<sub>6</sub>D<sub>6</sub>, and their dependence on constitution and solvent are discussed. The values of a certain mol. could be assumed to be composed additively of a number of parameters which characterize the substituents.  
 IT **35030-49-0**  
 RL: PRP (Properties)  
 (NMR of)  
 RN 35030-49-0 CAPLUS  
 CN Benzenepropanoic acid, α-phenyl-, methyl ester (9CI) (CA INDEX NAME)



- L15 ANSWER 49 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1973:110782 CAPLUS  
 DN 78:110782  
 TI Thallium in organic synthesis. XXXIV. Oxidations of acetylenes with thallium(III) nitrate (TTN)  
 AU McKillop, Alexander; Oldenziel, Otto H.; Swann, Brian P.; Taylor, Edward C.; Robey, Roger L.  
 CS Sch. Chem. Sci., Univ. East Anglia, Norwich/Norfolk, UK  
 SO Journal of the American Chemical Society (1973), 95(4), 1296-301  
 CODEN: JACSAT; ISSN: 0002-7863  
 DT Journal  
 LA English  
 AB The reactions of a variety of acetylenes with thallium(III) nitrate (TTN) have been examined, and the nature of the products has been found to depend on the solvent employed and the structure of the acetylene. Diarylacetylenes are converted into benzils in high yields on treatment with TTN in either aqueous acidic glyme or in MeOH; dialkylacetylenes gives acyloins in aqueous media and  $\alpha$ -methoxy ketones in MeOH; monoalkylacetylenes undergo degradation to carboxylic acids containing one C atom less than the starting material; and alkylarylacetylenes undergo smooth oxidative rearrangement in MeOH solution to give methyl  $\alpha$ -alkylarylacetates. All of the reactions proceed in high yield, and the specificity in oxidation is explained in terms of the mechanisms of the various reactions. Hydration of the C.tplbond.C bond does not occur to a significant extent.  
 IT **35030-49-0P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 35030-49-0 CAPLUS  
 CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester (9CI) (CA INDEX NAME)



- L15 ANSWER 50 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1972:45888 CAPLUS  
 DN 76:45888  
 TI Thallium in organic synthesis. XXVIII. Selective oxidation of acetylenes to carboxylic acids, acyloins, benzils, and arylacetic acids with thallium(III) nitrate  
 AU McKillop, Alexander; Oldenziel, Otto H.; Swann, Brian P.; Taylor, Edward C.; Robey, Roger L.  
 CS Sch. Chem. Sci., Univ. East Anglia, Norwich/Norfolk, UK  
 SO Journal of the American Chemical Society (1971), 93(26), 7331-3  
 CODEN: JACSAT; ISSN: 0002-7863  
 DT Journal  
 LA English  
 AB Reactions of a variety of acetylenes with thallium(III) nitrate were

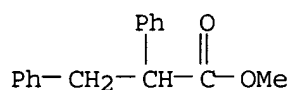
examined Diarylacetylenes may be converted into benzils in high yield, dialkylacetylenes give acyloins, monoalkylacetylenes undergo degradation to carboxylic acids containing 1 C atom less than the starting material, and alkylarylacetylenes undergo smooth oxidative rearrangement (in MeOH) to give Me  $\alpha$ -alkylarylacetates. All reactions proceed in high yield; the specificity in oxidation is explained in terms of the mechanisms of the various reactions. Hydration of the C.tplbond.C bond does not occur to a significant extent.

IT 35030-49-0P

RL: SPN (Synthetic preparation); PREP (Preparation)  
(preparation of)

RN 35030-49-0 CAPLUS

CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester (9CI) (CA INDEX NAME)



L15 ANSWER 51 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1971:476002 CAPLUS

DN 75:76002

TI Internal solvation effects on the conformation of acyclics

AU Auerbach, R. A.; Kingsbury, C. A.

CS Dep. Chem., Univ. Nebraska, Lincoln, NE, USA

SO Tetrahedron (1971), 27(11), 2069-77

CODEN: TETRAB; ISSN: 0040-4020

DT Journal

LA English

AB Expts. attempting to discern internal solvation (or H-bonding) of CO<sub>2</sub>H or CO<sub>2</sub>- by OH are outlined. In aqueous solns. little evidence for internal solvation exists. In basic MeOH this effect appears due to poor solvation by the solvent. The origin of anomalous coupling consts. in Me<sub>3</sub>C compds. is discussed.

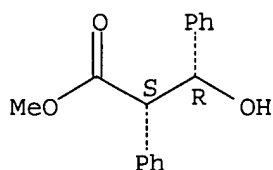
IT 17226-93-6 17226-94-7

RL: PRP (Properties)  
(conformation of, N.M.R. in relation to)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

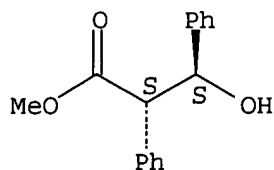
Relative stereochemistry.



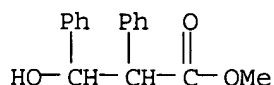
RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



- L15 ANSWER 52 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1971:435343 CAPLUS  
 DN 75:35343  
 TI Reformatsky reaction. Threo-erythro equilibration of bromozinc alkoxides  
 AU Bellassoued, Moncef; Couffignal, Rene; Gaudemar, Marcel  
 CS Lab. Synth. Organomet., Univ. Paris, Paris, Fr.  
 SO Comptes Rendus des Seances de l'Academie des Sciences, Serie C: Sciences  
 Chimiques (1971), 272(19), 1686-9  
 CODEN: CHDCAQ; ISSN: 0567-6541  
 DT Journal  
 LA French  
 AB Erythro-threo mixts. of  $\text{PhCR}_1(\text{OZnBr})\text{CHEtCO}_2\text{R}_2$  (I) ( $\text{R}_1 = \text{H}$  and  $\text{R}_2 = \text{Pr}$ )  
 (II) and I ( $\text{R}_1 = \text{Me}$ ,  $\text{R}_2 = \text{Et}$ ) (III) in different ratios were prepared by  
 treating the hydroxy esters with  $\text{BrZnCH}_2\text{CO}_2\text{Et}$  or by Reformatskii synthesis  
 from  $\text{PhCOR}_1$  and  $\text{BrZnCHEtCO}_2\text{R}_2$ . In  $\text{CH}_2(\text{OMe})_2$  or  $\text{Me}_2\text{SO}$  at  $-5^\circ$  or  
 $45^\circ$  for 0.5-6 hr, the mixture of secondary alcoholates of II did not  
 equilibrate; the III mixture (tertiary alcoholates), prepared by the 2nd  
 method, equilibrated slowly at  $45^\circ$  in  $\text{Me}_2\text{SO}$ , but not at  
 $-10^\circ$ . Thus, the Reformatskii reaction at low temperature is kinetically  
 controlled.  
 IT **93434-58-3DP**, Hydracrylic acid, 2,3-diphenyl-, methyl ester, zinc  
 complexes  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (preparation of)  
 RN 93434-58-3 CAPLUS  
 CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester (9CI)  
 (CA INDEX NAME)



- L15 ANSWER 53 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
 AN 1971:405086 CAPLUS  
 DN 75:5086  
 TI Thin-layer chromatography on silica gel as a method for assigning the  
 relative configurations to some aliphatic diastereomeric compounds  
 AU Palamareva, M.; Khaimova, M.; Stefanovski, Yu.; Viteva, L.; Kurtev, B.  
 CS Dep. Chem., Univ. Sofia, Sofia, Bulg.  
 SO Journal of Chromatography (1971), 54(3), 383-91  
 CODEN: JOCRAM; ISSN: 0021-9673  
 DT Journal  
 LA English  
 AB The configurations of the diastereoisomeric compds.  $\text{RCH}(\text{X})\text{CH}(\text{Y})\text{R}'$  (X and Y  
 are polar substituents and R and R' are Ph or m,p-dialkoxyphenyl) can be  
 determined by thin-layer chromatog. on silica gel. The erythro isomer always  
 exhibits a higher  $R_f$  value than the threo isomer, regardless of the  
 developing solvent polarity of the formation of an intramol. H-bond  
 between X and Y.  $R_f$  data are given for 37 diastereoisomeric pairs, and

10/446,319

the chromatog. behavior of the compds. is explained on the basis of the preferred conformations of the isomers. The compds. were detected either with Dragendorff reagent and Et<sub>2</sub>O-iodine or with H<sub>2</sub>SO<sub>4</sub>.

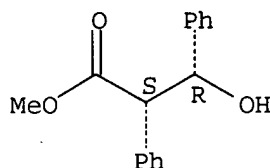
IT 17226-93-6 17226-94-7

RL: ANT (Analyte); ANST (Analytical study)  
(chromatog. of, thin-layer)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

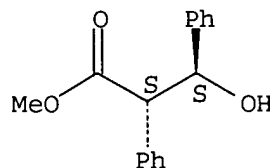
Relative stereochemistry.



RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 54 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1969:476047 CAPLUS

DN 71:76047

TI Nuclear magnetic resonance spectra, configuration and conformation of diastereomers 3-substituted 2,3-diphenylpropanoic acids and their methyl esters

AU Spasov, Stefan L.

CS Inst. Org. Chem., Sofia, Bulg.

SO Tetrahedron (1969), 25(16), 3631-38

CODEN: TETRAB; ISSN: 0040-4020

DT Journal

LA English

AB The N.M.R. spectra of the erythro and threo forms of compds. of the type PhCHAXCHBPhCO<sub>2</sub>R, where X = OH, OAc, NH<sub>2</sub>, NHMe, NHPh, NHCONH<sub>2</sub>, NHCONHAc, and R = H or Me, have been investigated in CDCl<sub>3</sub> and (CD<sub>3</sub>)<sub>2</sub>SO solns. at normal and in some cases also at higher temperature. The values of the vicinal coupling constant J<sub>AB</sub> are used to study the conformational equilibrium of the erythro and threo forms and its dependence on the solvent and temperature. The chemical-shift difference and the shape of the signals of some proton groups as R = Me and Ph are criteria permitting an unequivocal assignment of relative configuration of diastereomers of such type as well as their quant. determination in complex mixts.

IT 17226-93-6 17226-94-7

RL: PRP (Properties)  
(nuclear magnetic resonance of)

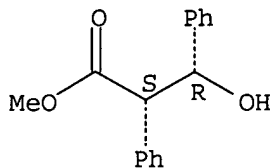
RN 17226-93-6 CAPLUS



10/446,319

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

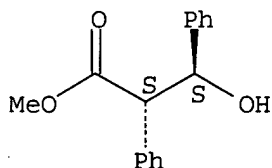
Relative stereochemistry.



RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 55 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1967:481706 CAPLUS

DN 67:81706

TI Stereochemistry of the Reformatskii reaction. II. Ir and N.M.R. spectra  
of  $\beta$ -hydroxy esters formed. Determination of their mixtures.  
Results

AU Canceill, Josette; Basselier, Jean J.; Jacques, Jean

CS College de France, Paris, Fr.

SO Bulletin de la Societe Chimique de France (1967), (3), 1024-30  
CODEN: BSCFAS; ISSN: 0037-8968

DT Journal

LA French

GI For diagram(s), see printed CA Issue.

AB cf. CA 66: 28487a. An ir and N.M.R. determination of the configuration of  
diastereoisomers of  $\beta$ -hydroxy esters (I and II) from the Reformatskii  
reaction of  $\text{ArRCO}$  with an  $\alpha$ -halo ester  $\text{BrR}_1\text{CHCO}_2\text{R}_2$  led to a  
discussion of the influence of chelation through intramol. H bonding and  
steric interactions in the conformational equilibrium of these compds. Risks  
are involved in the generalization of results of these spectroscopic  
methods applied to such problems. threo- and erythro-Hydroxy esters  
derived from benzaldehyde (I and II,  $\text{R} = \text{H}$ ) and from aromatic ketones ( $\text{R}$   
different from H) are discussed and the yields are tabulated. The preps.  
have been described (loc. cit.).

IT 17226-93-6 17226-94-7

RL: PRP (Properties)

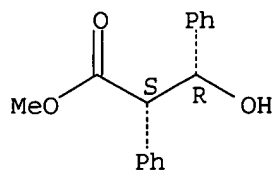
(configuration of, N.M.R. in relation to)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.

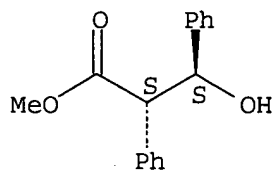
10/446,319



RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 56 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1963:481853 CAPLUS

DN 59:81853

OREF 59:15146f-h

TI Stereochemistry of the Reformatskii reaction

AU Canceill, Josette; Basselier, Jean Jacques; Jacques, Jean

CS College de France, Paris

SO Bulletin de la Societe Chimique de France (1963), (8-9), 1906-8

CODEN: BSCFAS; ISSN: 0037-8968

DT Journal

LA Unavailable

AB The stereochemistry of the Reformatskii reaction between PhCHO and BrCHRCO<sub>2</sub>R' was studied. The following PhCH(OH)CHRCO<sub>2</sub>R' were formed (R, R', % threo-compound in the mixture, m.p. of threo-compound, % erythro-compound in

the mixture, % yield of the reaction, and b.p. of the reaction mixture given):

Me, Me (I), 37  $\pm$  5, 48-50°, 63  $\pm$  5, 82, b3 122-7°; Et,

Me, 46  $\pm$  3, 42-3.5°, 54  $\pm$  3, 87, b2 141-2°; iso-Pr,

Me, 53  $\pm$  3, 36-7°, 47  $\pm$  3, 86, b3 124.5-6-5°; and Ph,

Et (II), 77  $\pm$  5, 79°, 23  $\pm$  5, 87, -. The configuration of

threo-II was determined by comparison with an authentic sample. The configuration of threo-I was proved by reduction of the compound with LiAlH<sub>4</sub> to a  $\beta$ -diol, which was identical with the compound obtained by transformation of trans- $\alpha$ -methylcinnamic acid into the corresponding ethylenic alc., followed by hydroboration of the alc. Nuclear magnetic resonance spectra were used to determine the configurations of the other stereoisomers. Possible intermediates in the Reformatskii reaction are discussed.

IT 17226-93-6, Hydracrylic acid, 2,3-diphenyl-, methyl ester, threo-

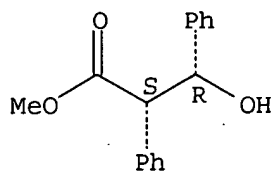
17226-94-7, Hydracrylic acid, 2,3-diphenyl-, methyl ester, erythro-

(preparation of)

RN 17226-93-6 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ S)-rel- (9CI) (CA INDEX NAME)

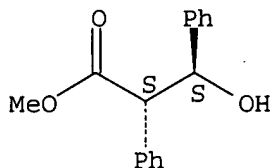
Relative stereochemistry.



RN 17226-94-7 CAPLUS

CN Benzenepropanoic acid,  $\beta$ -hydroxy- $\alpha$ -phenyl-, methyl ester,  
( $\alpha$ R, $\beta$ R)-rel- (9CI) (CA INDEX NAME)

Relative stereochemistry.



L15 ANSWER 57 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN

AN 1963:461744 CAPLUS

DN 59:61744

OREF 59:11299d-h,11300a-d

TI Analgesics. Absolute configuration of  $\alpha$ -(+)-4-dimethyl-amino-1,2-diphenyl-3-methyl-2-propionybutane, d-propoxyphene

AU Sullivan, H. R.; Beck, J. R.; Pohland, A.

CS Eli Lilly &amp; Co., Indianapolis, IN

SO Journal of Organic Chemistry (1963), 28(9), 2381-5

CODEN: JOCEAH; ISSN: 0022-3263

DT Journal

LA Unavailable

GI For diagram(s), see printed CA Issue.

AB  $\alpha$ -(+)-Me<sub>2</sub>NCH<sub>2</sub>CHMeCPh(CH<sub>2</sub>Ph)O<sub>2</sub>CEt.HCl (I) (15 g.) and 135 mL. 5N HCl was refluxed 2 h. and concentrated to dryness to give 8.5 g. (--)Me<sub>2</sub>NCH<sub>2</sub>CHMeCPh:CHPh.HCl (II), m. 189-90° (MeOH-EtOAc), [ $\alpha$ ] (all 25/D) -33.7° (c 1, H<sub>2</sub>O). Similarly, 50 g.  $\alpha$ -(--) isomer of I gave 21 g. II (--) isomer (III), m. 189-90°, [ $\alpha$ ] 34.3° (c 1, H<sub>2</sub>O). Into 25 g. II, 150 mL. MeOH, and 1100 mL. EtOAc at -20° was passed O<sub>3</sub> in excess; the whole added to 250 mL. ice-H<sub>2</sub>O, kept overnight, the MeOH and EtOAc distilled in vacuo, the aqueous solution washed with Et<sub>2</sub>O (concentration of these gave BzH), the aqueous solution treated with excess aqueous NH<sub>3</sub>, extracted with Et<sub>2</sub>O, and the Et<sub>2</sub>O, exts.

dried and treated with dry HCl gave 8 g. (--)Me<sub>2</sub>NCH<sub>2</sub>CHMeBz.HCl (IV), m. 153-4° (MeOH-EtOAc), [ $\alpha$ ] -47° (c 1, H<sub>2</sub>O). To 11.8 g.

IV, 130 g. anhydrous Na<sub>2</sub>HPO<sub>4</sub>, and 300 mL. CH<sub>2</sub>Cl<sub>2</sub> at 0-5° was added CF<sub>3</sub>-CO<sub>3</sub>H [from 50.8 g. (CF<sub>3</sub>CO)<sub>2</sub>O, 8.2 mL. 90% H<sub>2</sub>O<sub>2</sub>, and 100 mL. CH<sub>2</sub>Cl<sub>2</sub>, at 0°]; the whole stirred 1 h. (temperature rise to 10°), the solid mixture kept 4 h. at room temperature, 200 mL. H<sub>2</sub>O added, the CH<sub>2</sub>Cl<sub>2</sub> phase separated,

evaporated to dryness, the residue dissolved in 100 mL. 2N HCl, washed, treated with excess concentrated aqueous NH<sub>3</sub> and extracted with Et<sub>2</sub>O and the dried Et<sub>2</sub>O

solution treated with dry HCl gave 3.8 g. (--)Me<sub>2</sub>NCH<sub>2</sub>CHMeOBz.HCl, m. 160-1° (MeOH-EtOAc), [ $\alpha$ ] -62.8° (c 1, H<sub>2</sub>O).

D(-)-H<sub>2</sub>NCH<sub>2</sub>CH-(OH)Me.HCl (2.7 g.), 1.6 g. HCO<sub>2</sub>Na, 8.3 g. 100% HCO<sub>2</sub>H, and

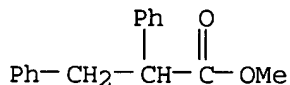
6.7 g. 37% HCHO were refluxed 14 h. to give 3.13 g. hygroscopic D(--)-Me<sub>2</sub>NCH<sub>2</sub>CH(OH)Me.HCl (V), [α] -44.5° (c 1, MeOH); V, 7.1 g. BzCl, 25 mL. Et<sub>3</sub>N, and 80 mL. CHCl<sub>3</sub> refluxed 1 h. gave 4.2 g. Me<sub>2</sub>NCH<sub>2</sub>CH(OBz)Me, HCl, m. 160-1° (MeOH-EtOAc), [α] -63.3° (c 1, H<sub>2</sub>O). I (370 g.), 4.5 l. MeOH, and 650 mL. 30% H<sub>2</sub>O<sub>2</sub> kept 48 h. at room temperature, the whole evaporated to dryness, 0.4 g. PtO<sub>2</sub> added (caution), filtered, the filtrate evaporated to dryness, and the residue in 2 l. EtOAc treated with 40 g. HCl in 1 l. EtOAc gave 370 g. I N-oxide-HCl (VI), m. 186-7° (MeOH EtOAc), [α] 20.2° (c 2, MeOH); VI in 1500 mL. H<sub>2</sub>O saturated with K<sub>2</sub>CO<sub>3</sub> and extracted with Et<sub>2</sub>O gave 320 g. N-oxide (VII); VII heated slowly at 0.5 mm. to 140°, kept 0.5 h. at 140°, the whole cooled, dissolved in Et<sub>2</sub>O and the Et<sub>2</sub>O solution washed, dried, concentrated and distilled gave 200 g. (+)-CH<sub>2</sub>:CMeCPh(CHPh)O<sub>2</sub>CEt (VIII), b<sub>0.6</sub> 152°, n<sub>25D</sub> 1.5466, [α] 97° (c 1.3, Me<sub>2</sub>CO) (the cold trap from the pyrolysis contained Me<sub>2</sub>NHOH). VIII (45 g.) in 1 l. EtOAc at -50° and O<sub>3</sub> to blue color, the whole poured into ice-H<sub>2</sub>O, kept overnight, dilute aqueous NaHSO<sub>3</sub> added, dropwise, to neg. starch-iodide test, the EtOAc solution separated, washed and dried gave 41 g. (+)-AcCPh-(O<sub>2</sub>CEt)CH<sub>2</sub>Ph (IX), b<sub>0.5</sub> 156-7°, [α] 182.7° (c 1.1, Me<sub>2</sub>CO). IX (32.5 g.), 1 l. 5N HCl, and 1 l. EtOH refluxed 20 h. gave 21.9 g. (+)-AcC(OH)PhCH<sub>2</sub>Ph (X), b<sub>0.5</sub> 148°, n<sub>25D</sub> 1.5660, [α] 141° (c 1.3, EtOH). To 60 g. X in 200 mL. glacial AcOH was added, dropwise, 45 g. Br in 100 mL. glacial AcOH, and the whole stirred 1 h. and evaporated to dryness in vacuo; the residue in 600 mL. C<sub>5</sub>H<sub>5</sub>N heated 1 h. at 100°, the whole concentrated, and the residue and 60 g. NaOH in 900 mL. H<sub>2</sub>O heated 2 h. at 100° gave 20 g. (-)-PhCH<sub>2</sub>C(OH)PhCO<sub>2</sub>H (XI), m. 145-6° (aqueous EtOH), [α] 13.7° (c 3.5, EtOH). XI (18 g.), 40 g. Ag<sub>2</sub>O and 200 mL. MeI refluxed and stirred 3 h. gave 15 g. Me ester (XII), m. 102-3° (Et<sub>2</sub>O-petr. ether), [α] -32.4° (c 3.4, CHCl<sub>3</sub>). Absolute EtOH, (200 mL.) and 60 g. freshly prepared W-2 Raney Ni, shaken first with 40 lb./in.2 H, 8 g. XII added, and the whole refluxed 8 h., filtered, the residue from the concentration of the filtrate dissolved in 40 mL. petr. ether, 1.4 g. XII which separated, filtered off, the filtrate evaporated and the residue distilled gave 6 g. (+)-PhCH<sub>2</sub>CHPhCO<sub>2</sub>Me (XIII), b<sub>0.2</sub> 123-4°, n<sub>25D</sub> 1.5518, [α] -90.5° (c 4.6, CHCl<sub>3</sub>). To 1.7 g. LiAlH<sub>4</sub> in 110 mL. anhydrous Et<sub>2</sub>O was added 10.2 g. XIII in 40 mL. Et<sub>2</sub>O, dropwise, and the whole refluxed 3 h. to give 7.8 g. (+)-PhCH<sub>2</sub>CHPhCH<sub>2</sub>OH, b<sub>0.2</sub> 128°, n<sub>25D</sub> 1.5742, [α] 76.3°; tosylate (XIV) (prepared in C<sub>5</sub>H<sub>5</sub>N), m. 89-90° (Me<sub>2</sub>CO-petr. ether), [α] 40.2° (c 2.4, Me<sub>2</sub>CO). XIV (7.4 g.), 16.5 g. NaI, and 200 mL. anhydrous Me<sub>2</sub>CO refluxed and stirred 20 h. gave 6.35 g. (+)-PhCH<sub>2</sub>CHPhCH<sub>2</sub>I (XV), m. 54-5° (petr. ether), [α] 16.6° (c 3.9, EtOH); XV, 3.0 g. NaHCO<sub>3</sub>, 150 mL. absolute EtOH, and 5 g. Pd-C hydrogenated 10 h. at 25° gave 2.6 g. (+)-PhCH<sub>2</sub>CHPhMe, b<sub>0.3</sub> 85°, n<sub>25D</sub> 1.5558, [α] 76.7° (c 2.3, CHCl<sub>3</sub>). (-)-threo-PhCH(OH)CHPhMe (1.4 g.) added to 100 mL. absolute EtOH and 30 g. W-2 Raney Ni, shaken first in H as above, and the whole refluxed 8 h. gave 1.15 g. D(-)-PhCH<sub>2</sub>CHPhMe, b<sub>0.5</sub> 88°, n<sub>25D</sub> 1.5553, [α] -76.3° (c 2.2, CHCl<sub>3</sub>). The absolute configuration of I is (2S,3R).

IT 35030-49-0, Propionic acid, 2,3-diphenyl-, methyl ester (preparation of)

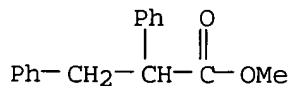
RN 35030-49-0 CAPLUS

CN Benzenepropanoic acid, α-phenyl-, methyl ester (9CI) (CA INDEX NAME)

10/446,319



L15 ANSWER 58 OF 58 CAPLUS COPYRIGHT 2005 ACS on STN  
AN 1962:462469 CAPLUS  
DN 57:62469  
OREF 57:12373g-h  
TI Relative signs of geminal and vicinal proton-proton coupling constants in  $\alpha,\beta$ -diphenylpropionic acid and its methyl ester  
AU Fraser, Robert R.  
CS Univ. Ottawa  
SO Canadian Journal of Chemistry (1962), 40, 1483-9  
CODEN: CJCHAG; ISSN: 0008-4042  
DT Journal  
LA Unavailable  
AB Since theoretical calcns. of vicinal coupling consts. and geminal coupling consts. predicted the same sign as long as the bond angle between the geminal hydrogens is less than  $125^\circ$ ; it has now been shown that geminal and vicinal consts. are opposite in sign in  $\alpha,\beta$ -diphenylpropionic acid (I) and its Me ester (II). The spectrum of I measured at 60 Mc./sec. shows 11 transitions. The spectrum of II shows 13. A tabulation of intensities and spectra at other frequencies is listed.  
IT **35030-49-0**, Propionic acid, 2,3-diphenyl-, methyl ester  
(nuclear magnetic resonance and spin-spin coupling of)  
RN 35030-49-0 CAPLUS  
CN Benzenepropanoic acid,  $\alpha$ -phenyl-, methyl ester (9CI) (CA INDEX NAME)



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